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# Small-Scale Safety and Thermal Testing of Improvised Explosives—Correlation of Results from a Multiple-Laboratory Proficiency Test

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**Abstract.** Over 30 issues have been identified that indicate standard test methods may require modification when applied to home made or improvised explosives (HMEs) to derive accurate sensitivity assessments by small-scale safety and thermal (SSST) testing. These results come from a round-robin type proficiency test conducted among five explosives testing laboratories for the Integrated Data Collection Analysis (IDCA) Program sponsored by the Department of Homeland Security. The participants had similar equipment, usually differing by vintage. This allowed determining how each participant performed on a specific material and how this performance differed from the average. Some general trends observed for each series of tests include: 1) Drop hammer—LLNL usually found the materials less reactive than the average at low drop heights and LANL usually found the materials less reactive than the average at high drop heights; 2) Friction— LLNL found the materials less sensitive than the average; 3) Electrostatic discharge (ESD)—NSWC IHD usually found the materials less sensitive than the average; 4) Constant heating rate differential scanning calorimetry (DSC)—very reproducible for all the participants. In addition, the standard, RDX, was tested multiple times throughout the proficiency test by all the participants. This provided a very large set of data to apply statistical analysis typical for this type of testing. Application showed there were statistical differences among the performers due to sandpaper type (impact), operator and detection method (impact, friction, ESD), humidity (ESD), age of equipment (ESD) and sample pan type (DSC). These results will be discussed in this report in terms of the how accurate are SSST testing data with respect to inter- and intra-laboratory testing.

#### Introduction

One of the first steps in establishing safe handling procedures for explosives is small-scale safety and thermal (SSST) testing. 1,2 To better understand the response of home made or improvised explosives (HMEs) to SSST testing, 16 HME materials were compared to three standard military

explosives in a proficiency-type round robin study among five laboratories, two U.S. Department of Defense and three U.S. Department of Energy, sponsored by the Department of Homeland Security, Science & Technology Directorate, Explosives Division. The testing matrix has been designed to address problems encountered with improvised

materials: powder mixtures, liquid suspensions, partially wetted solids, immiscible liquids, and reactive materials. All testing materials and/or precursors came from the same batch distributed to each of the participants and were handled, pretreated, and mixed by the standardized procedures.

Over 30 issues have been identified that indicate standard test methods may require modification when applied to HMEs to derive accurate sensitivity assessments.<sup>3,4</sup> The recommendations for modification of testing are: 1) develop new sampling methods that guarantee obtaining a representative sample, particularly for very small samples of mixtures, samples that have a volatile component, and samples that have large mismatch of particle sizes; 2) carefully assess particle-size distributions of mixtures, as particle size affects most measurements; 3) recognize that safety testing is linked to the handling conditions (so safety testing conditions must reflect the operation that is being assessed); 4) recognize that relative sensitivity to a standard can change when testing conditions are altered, and that testing may not reflect the true sensitivity of the material for specific application; 5) develop new methods to test liquids, specifically handling the volatility issue and standards; 6) develop instrument-based detection to lessen the reliance on observation.

These results will be discussed in terms of the how accurate are SSST testing data with respect to inter- and intra-laboratory testing.

#### **Experimental**

The experimental methods have been reviewed in detail elsewhere. 5.6 Briefly; the testing included impact (Type 12 drop hammer), friction (German Bundesanstalt für Materialprüfung—BAM and Allegany Ballistics Laboratory—ABL methods), electrostatic discharge, ESD (ABL and custom built), and thermal (constant heating rate differential scanning calorimetry—DSC). All participating laboratories had some version of this suite of testing instruments.

The source and preparation of the materials used in the proficiency test have also been reviewed previously. All pretreatments and mixing followed IDCA procedures.<sup>6,7</sup> Specifically, drying was at 60°C for 16 hours, then the materials were cooled and stored in a desiccator.

#### Results

Table 1 shows the materials studied, the abbreviation found throughout the text and the physical form. The variety and complexity of the forms of the mixtures and compound provided many challenges for testing.

Table 1. IDCA mixtures and pure materials formulations

Torritalations			
Material <sup>a</sup>	ID	Form <sup>b</sup>	
KCIO <sub>4</sub> /AI	KP/AI	Dry powder	
KCIO <sub>4</sub> /C <sup>c</sup>	KP/C	Dry powder	
KClO₄/dodecane	KP/D	Wet powder	
KClO₃/dodecane	KC/D	Wet powder	
KClO₃/sugar <sup>d</sup>	KC/Sugar 100	Dry powder	
KClO₃/sugar <sup>e</sup>	KC/Sugar AR	Dry powder	
NaClO₃/sugar <sup>d</sup>	SC/Sugar	Dry powder	
AN <sup>f</sup>	AN	White powder	
Bullseye® gunpowder	GP	Black powder	
AN/Bullseye® gunpowder	AN/GP	Gray powder	
UNi/Al <sup>g</sup>	UNi/Al	Dry powder	
UNi/Al/S	UNi/AI/S	Dry powder	
H <sub>2</sub> O <sub>2</sub> /cumin <sup>h,i</sup>	HP/Cumin	Viscous paste	
H <sub>2</sub> O <sub>2</sub> /nitromethane <sup>j</sup>	HP/NM	Miscible liquid	
H₂O₂/flour <sup>h,k</sup>	HP/Flour	Sticky paste	
H <sub>2</sub> O <sub>2</sub> /glycerol <sup>h</sup>	HP/Glycerol	Miscible liquid	
HMX Grade B	HMX	Powder	
RDX Type II Class 5	RDX	Powder	
PETN Class 4	PETN	Powder	

<sup>a</sup> Mixture or pure material, <sup>b</sup> observed physical form, <sup>c</sup> activated charcoal (Darco), <sup>d</sup> icing sugar + -100 mesh KClO<sub>3</sub>, <sup>e</sup> icing sugar + as received KClO<sub>3</sub>, <sup>f</sup> ammonium nitrate, <sup>g</sup> Urea nitrate, <sup>h</sup> 70 percent H<sub>2</sub>O<sub>2</sub>, <sup>i</sup> cuminum cyminum, <sup>j</sup> 90 percent H<sub>2</sub>O<sub>2</sub>, <sup>k</sup> chappati, <sup>l</sup> standard

Generalized Comparison of Results among Participants

The five-laboratory team helps answer the question: are there differences in SSST testing results among participants for a specific material?

Impact sensitivity. Fig. 1 shows a graph of the impact data from the individual participants compared to the average of all the participants for each material. The values are the DH<sub>50</sub>, in cm, by a modified Bruceton method, 8 load for 50 percent probability of reaction. The red line is the average data and the symbols are Individual laboratory data.

The results in Fig. 1<sup>3,4</sup> are for the 0 to 50 cm range and show: LLNL (red circles) values mostly are above the average line for sensitivities below DH<sub>50</sub> of 50 cm; LANL (blue squares) values gen-

erally are below the average line for sensitivities below DH<sub>50</sub> of 50 cm; IHD generally tracks LANL values, but show slightly higher sensitivity; AFRL values generally reports the highest sensitivity of the all the participants for a specific material. The 90 to 170 cm drop height range (not shown) indicates: LLNL and AFRL are below the average; LANL values are above the average: IHD values do not exhibit a trend.

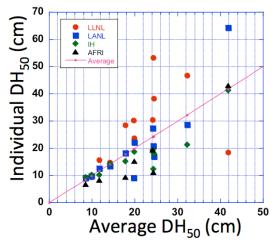


Fig. 1. Impact sensitivity data by modified Bruceton method ( $DH_{50}$  in cm) for the average of all participants vs. the average of each of the participants in range of 0 to 40 cm.

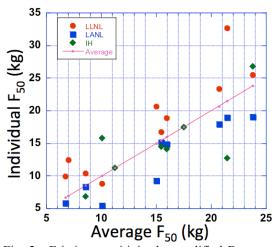


Fig. 2. Friction sensitivity by modified Bruceton method ( $F_{50}$  in kg) for the average of all participants vs. the average of each of the participants taken on BAM testing equipment.

Friction sensitivity. Friction sensitivity was measured in the proficiency test by testing with BAM and ABL equipment. Friction testing results, F<sub>50</sub>, are reported here with the BAM equipment. F<sub>50</sub>, in kg, is by a modified Bruceton method, load for 50 percent probability of reaction. The red line is the average of all the data, in kg, and the symbols are the individual lab data. The results for the ABL equipment will be reported below with a comparison analysis of these materials on both the ABL and BAM equipment.

The results in Fig. 2 show: LLNL (red dots) always derives a value for  $F_{50}$  above the corresponding average value for each of the materials (except in one case); LANL (blue squares) always derives a value for  $F_{50}$  below the corresponding average value for each of the materials; IHD (green diamonds) values tend to be around the corresponding average values. LLNL finds the materials to be less sensitive.

Spark Sensitivity. In the proficiency test, two ESD systems were used, the commercially available ABL system (differing vintages) and a custom built system by LLNL. Except where noted, all the data compared below were derived from comparable ABL systems. Fig. 3 shows the data. The red line is the average data and the symbols are Individual laboratory data.

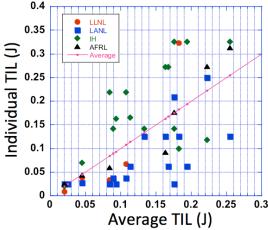


Fig. 3. ESD sensitivity data (TIL in J) for the average of all participants vs. the average of each of the participants taken on ABL testing equipment.

The data are represented by TIL<sup>9</sup>, which is the load (joules) at zero reactions out of 20 or fewer

trials with at least one reaction out of 20 or fewer trials at the next higher energy level. Individual data of each participant are represented vs. average data of all the participants (1:1 is red line). LLNL (red dots) and AFRL (black triangles) have limited data sets with the ABL device. In general, IHD (green diamonds) report lower sensitivities compared to LANL (blue squares).

Comparison of BAM Friction and ABL Friction Results

In the proficiency test, friction sensitivity was measured by both the BAM and ABL methods. Usually, a testing laboratory has one or the other instruments. However, IHD had both types of equipment allowing for a direct comparison of both types of data. This facilitated an answer to the question is there a translation function between the two techniques?<sup>10</sup>

The architectural designs of ABL friction and BAM friction testing equipment are vastly different and hence the response to the various HMEs is different. The ABL is more like a "nip" and BAM is more "plow like". Fig. 4 compares the operational parameters of the two methods, accentuating the differences in the mechanisms by which the friction insults are applied.

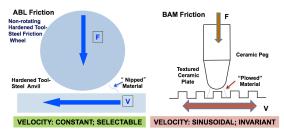


Fig. 4. Diagrams of ABL and BAM Friction action during testing

The ABL method has only hardened steel surfaces, while the BAM method uses porous ceramics. The insult point for the ABL method is a nipped or pinched area between non-porous two steel surfaces. While for the BAM method, the sample is plowed over a porous surface with the use of a ceramic peg. In both cases, the support surface moves, but this motion is different in the two cases. It is also important to remember that the ABL applies force using pressure-regulated action and BAM applies force using weight regu-

lated action, so the response levels are in psig and kg, respectively.

Table 2 lists the average data for the  $F_{50}$  and TIL determinations by the ABL and BAM methods. Conditions of the measurements have been reported elsewhere. The method for determining the average has been delineated previously.

Table 2.  $F_{50}$  and TIL values by ABL and BAM Friction Methods

Material	ABL TIL, psig; F <sub>50</sub> , psig	BAM TIL, kg; F <sub>50</sub> , kg
KP/AI	< 30; 51	16.5; 26.8
KP/C	112; 281	> 36.7; ND
KP/D	350; 717	33; > 36.7
KC/D	135; 498	16.5; 26.8
KC/Sugar 100	30; 42	2.3; 4.4
KC/Sugar AR	123; 150	3.2; 3.6
SC/Sugar	225; 447	4.4; 15.8
AN	385; ND	36.7; > 36.7
GP	ND; 316	13.9; > 36.7
AN/GP	76.6; 159	12.2; 12.7
UNi/Al	217; 555	> 36.7; ND
UNi/AI/S	217; 376	> 36.7; ND
HP/Cumin	> 1000; ND	8.6; 11.2
HP/NM	> 1000; ND	> 36.7; ND
HP/Flour	> 1000; ND	11.4; ND
HP/Glycerol	> 1000; ND	11.8; 17.5
HMX	45; 112	8.6; 14.1
RDX Set 1	55; 141	15.1; ND
RDX Set 2	92; 207	11.8; 27.8
RDX Set 3	92; 123	11.4; 19.3
RDX Set 4	75; 160	11.8; 17.5
PETN	7.7; 42	4.3; 6.9

1000 psig is upper limit for ABL method; 36.7 kg is upper limit for BAM method

The differences in design of the two methods are evident in some of the materials. For example, the HP/fuel mixtures exhibit no sensitivity in the ABL method, but have various level of sensitivity by the BAM method. The UNi mixtures show the opposite trend exhibiting no sensitivity by the BAM method but reasonable sensitivity by the ABL method.

The relative ordering of the  $F_{50}$  or TIL values of the sensitivities of the materials highlight the differences (and similarities) of the two testing methods. If the sensitivity of a specific material is compared relative to a well-characterized standard, then the almost impossible task of comparing sensitivities in kg to sensitivities in psig at a specific velocity is somewhat overcome. For TIL and  $F_{50}$  for the two methods, ABL and BAM, KC/Sugar

and PETN are generally on the top of the list for friction sensitivity. HMX is rated relatively less, but still near the top of the list for relative sensitivity. On the opposite end of the sensitivity scale, UNi mixtures tend to exhibit little or no friction sensitivity for both the methods.

Many of the other materials exhibit one type of behavior for one method, and the opposite behavior for the other method. Examples of these are KP/Al, KP/C, and SC/Sugar.

Liquids and pastes tend to be less sensitive on the ABL method compared to the BAM with exception of HP/NM mixture. This could be attributed to the miscibility of nitromethane in hydrogen peroxide.

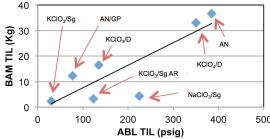


Fig. 5. Friction sensitivity by TIL assessment by the ABL (x-axis) and the BAM (y-axis) methods

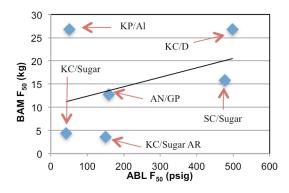


Fig. 6. Friction sensitivity by  $F_{50}$  assessment by ABL (x-axis) and the BAM (y-axis) methods

In attempt to determine if there is a direct correlation between the two methods the TIL data for the materials are shown in Fig. 5 and for the  $F_{50}$  data in Fig. 6. In both figures, the x-axis represents the ABL data values, and the y-axis represents the corresponding BAM data values.

Clearly there is no correlation of the data between the two testing methods. Dividing the data into subgroups does not provide any correlations (HMEs, TIL  $R^2 = 0.5372$ ,  $F_{50}$   $R^2 = 0.16708$ ).

# Comparison of Statistical Analysis of RDX

In the proficiency test, RDX Type II Class 5 was used as the primary standard. As a result, the material was examined several times throughout the testing. This provides a significant amount of data on the same material to calculate some statistics that can help answer the question what is the margin of error for a specific determination of RDX? These values can be used as a basis for the margin of error on other materials that statistics cannot be done on because of time and material limitations.

Equivalency of RDX drop hammer (DH50) data. Fig. 7 shows box plots<sup>11</sup> of the impact data taken for RDX during the proficiency test. The data are grouped by participant (LLNL, LANL, IHD, AFRL, and SNL), sandpaper type (180 is 180-grit garnet, 150 is 150-grit garnet; 120 is 120-grit Si/C), and whether the data were reduced by the Bruceton<sup>8</sup> or Never<sup>12</sup> (B or N, respectively) methods. The colored boxes are 50 percent of each data set; the mean is the center of the box; the median is the line in the box; and the range is the vertical bar. The full sets of testing variables are sandpaper, striker weight, temperature, humidity, detection method, and operator. The invariables are the source of the RDX (all from the same batch), the drying procedure and the mixing procedure.

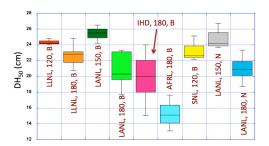


Fig. 7. Box plot of the DH<sub>50</sub> values grouped by participant sandpaper grit size and data reduction method

Visual inspection suggests that the results range from symmetric to skewed and that a subset or subsets of the different groupings are likely in agreement with each other, based on overlap of the shaded regions and to some extent the max/min bars. The AFRL 180 data appears to be significantly separated from the rest.

Further analyses were conducted to verify statistically this observation (AFRL 180 data is separated). Analysis of variants or ANOVA<sup>13,14</sup> analysis yielded a p-value of 0.000, indicating at least one of the data sets represented in Fig. 7 is statistically different than the others and there is less than 0.1 percent chance that this assessment is in error.

Although the p-value indicates there is an outlier, it does not indicate which one. Further analysis by Tukey and Fisher methods<sup>13,15</sup> allow subgrouping to better identify the outlier. The result of these subgrouping is discussed elsewhere<sup>11</sup>, but both methods show the AFRL 180 data set is categorized alone into a subgroup, further substantiating the ANOVA results above.

Further analyses data obtained during the proficiency test on RDX<sup>11</sup> show, for DH<sub>50</sub>, the expected results are a mean value of 21.5 cm with a 27 percent variability, and for BAM  $F_{50}$ , a mean value of 21.0 kg and a 40 percent variability.

Correlations with testing variables. In SSST testing, the environmental variables are often attributed for the differences in results on the same material. As a partial test of this theory, the RDX results were parameterized with a limited number of obvious variables.

Fig. 8 shows the impact data (DH<sub>50</sub>, in cm) as a function of sandpaper type (noted as grit size), testing room temperature, testing room relative humidity, and striker mass. Other than possible sandpaper effects, the graphs offer no correlations.

Fig. 9 shows a similar examination of the results obtain using the BAM friction instrument using a modified Bruceton ( $F_{50}$ , in kg) or the threshold initiation level (TIL, in kg) analysis methods for testing room temperature, and testing room relative humidity. There is a possible correlation with humidity for the  $F_{50}$  data (from IHD, 40% RH).

Many comparisons have been reported on the effect of sandpaper on impact sensitivity.<sup>3</sup> As well, studies on the role of grit on non-shock initiated reactions indicate that grit size and hardness are important parameters for initiation.<sup>17</sup> Further work has been conducted on determining the extent of the sandpaper effect by varying particle size

of the energetic material and the grit size and composition of the sandpaper. No further work on determining the relationship of humidity to BAM and ABL friction measurements is planned.

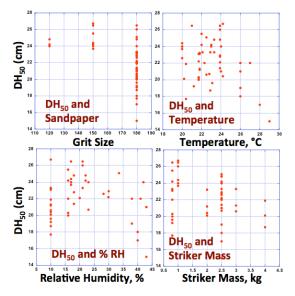


Fig. 8. Comparison of RDX Class 5 Type II DH<sub>50</sub> with various method and environment variables

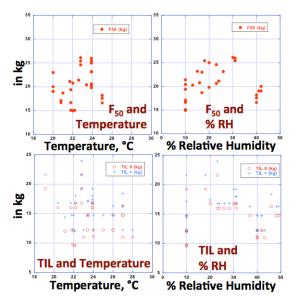


Fig. 9. Comparison of RDX Class 5 Type II  $F_{50}$  and TIL with various method and environment variables.

ESD threshold values. ESD provides coarse data because of the ways the energy levels are set (each participant sets discrete levels differently. This presents many repetitive values and clustering, but ANOVA analysis cannot be applied, so this data set is handled differently than above.

Fig. 10 shows the ESD data sets determined as TIL and the level above TIL (TIL+, at least one positive out of 10 or 20 trials). The data from IHD do not overlap with the data from LLNL and LANL. This has been attributed to the much higher amount of humidity at IHD, compared to the other testing laboratories. The TIL range is 0.025 to 0.095 J, average 0.046 J with a > 50 percent variability. The TIL+ range is 0.0625 to 0.165 J with an average of 0.091J and a variability that cannot be calculated because some results fall outside of experimental range.

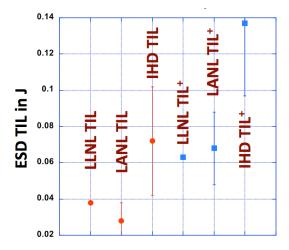


Fig. 10. Average TIL and TIL+ values for RDX Class 5 Type II ESD results. LLNL results using the custom instrument with a  $510-\Omega$  resistor are not included.

Thermal data for RDX. Thermal sensitivity was measured by differential scanning calorimetry (DSC) during the proficiency test. The default conditions were a 10°C/min heating rate, TA Instruments pinhole vented sample holder, and approximately 40 to 500°C heating range.

For RDX, the proficiency test yielded 46 different data sets, with 3 different sample holder types (2 sealed and 1 open). Fig. 11 shows a comparison of the RDX examined in the standard sam-

ple holder and a specialized high pressure SWISSI sample holder.

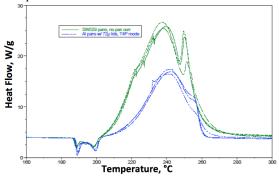


Fig. 11. Example DSC scans of RDX Class 5 Type II in typical pinhole hermetic pans (blue lines) and one type of sealed pan (green Lines).

The details of the comparison of the sample holders can be found elsewhere.<sup>5,11</sup> The results show the measurements with the sealed sample holders indicate higher enthalpies of decomposition because they do not allow gas to escape during heating. For example, the exothermic broad maximum in Fig. 11 is around 4000 J/g and 2000 J/g for the SWISSI and pinhole sample holders, respectively. The pinhole sample holders allow gas to escape at a controlled rate that removes heat from the system, lowering the total observed enthalpy. This also affects the maximum or minimum temperatures of these features, but to a lesser extent. For example, the T<sub>max</sub> range of the broad exothermic feature in Fig. 11 is 237 to 242 °C and 239 to 244 °C, for SWISSI and pinhole sample holders, respectively.

### Discussion

Generalized Comparison of Results Among Participants

The IDCA found when applying standard SSST testing techniques to HMEs, modification of procedures might be required before meaningful data can be derived. Below are results from testing a specific material or materials that can be generalized to issues that need consideration before testing any HME.

• Pure solids—sandpaper selection can affect impact sensitivity results so grit size, sample preparation and particle size must be considered;

- Solid-solid mixtures—same sandpaper issues for impact sensitivity; in addition, obtaining a representative sample and particle size mismatch have to be considered in determining reactivity in all tests, especially DSC;
- Solid-liquid mixtures—volatility of components, solid particle issues, and obtaining a representative sample must be considered for all tests;
- Liquid-liquid mixtures—volatility of components and mixing are important considerations in obtaining a representative sample for all tests;
- Relative sensitivity compared to standards—sandpaper affects both sample and standard, but not necessarily the same way; something can be sensitive with one sandpaper and not sensitive with another in impact testing;
- Absolute sensitivity—operator subjectivity, testing environment and choice of testing parameters

can affect the absolute sensitivity that can question the authenticity of the absolute number;

• Specific cases—certain materials have shown that SSST testing may not be possible because of the physical nature of the material.

Comparison of Statistical Analysis of RDX Results

The RDX standard was tested multiple times during the proficiency test by all the participants yielded enough data to make set expectations of future results based on statistical analysis. Table 3 shows these expected results and variation for RDX for all the SSST testing. The table also includes suggested sources for the variation, both environmental and other. A full discussion of the results can be found elsewhere. <sup>11</sup>

Table 3. Ranges of DSC Parameters for RDX Class 5 Type II

Parameter <sup>1</sup>	Pinhole Old	Pinhole New	Sealed LLNL	Sealed IHD
Endothermic Onset, °C	187.3-187.8	187.4-188.6	187.3-187.8	186.1-187.8
Range (Average)	(187.7 ± 0.2)	(188.0 ± 0.2)	(187.6 ± 0.2)	(187.4 ± 0.6)
Endothermic Minimum, °C	188.3-189.2	188.7-189.9	188.3-189.1	188.3-190.0
Range (Average)	(188.9 ± 0.3)	(189.4 ± 0.3)	(188.8 ± 0.3)	(189.3 ± 0.8)
Endothermic Minimum, °C	198.8-200.0	198.6-200.8	198.8-199.4	198.1-199.8
Range (Average)	(199.2 ± 0.3)	(199.9 ± 0.5)	(199.0 ± 0.2)	(199.1 ± 0.8)
Endothermic Enthalpy, J/g	126-181	92-146	114-144	92-123
Range (Average)	(142 ± 15)	(128 ± 14)	(133 ± 9)	(102 ± 11)
Exothermic Onset, °C Range <sup>2</sup>	203-219	201-225	203-220	209-215
Exothermic Maximum, °C	238.7-243.5	239.8-244.2	230.6-244.0	237.4-241.9
Range (Average)	(241.6 ± 1.4)	(242.3 ± 1.0)	(235.3 ± 3.9)	(239.8 ± 1.8)
Exothermic Enthalpy, J/g	1890-2432	1947-2385 <sup>3</sup>	2003-3805	4203-4662
Range (Average)	(2244 ± 177)	(2174 ± 120)	(3108 ± 495)	(4423 ± 179)

<sup>1.</sup> Onset is the beginning of the maximum or minimum as automatically identified by the equipment, endothermic min. is the minimum temperature of the endothermic feature, endothermic enthalpy is the overall enthalpy of the two overlapping endothermic features, exothermic max. is the maximum of the exothermic feature; 2. Range only because the transition between the endothermic and exothermic features overlap; 3. Two values from IHD Set 2 discarded due to sample holder rupturing during experiment

#### Conclusions

The conclusions from the analysis of results from the IDCA proficiency test are best understood by listing some general recommendations for HME testing. These recommendations are the following:

• Develop new sampling methods that guarantee obtaining a representative sample particularly for very small samples of mixtures, samples that have

volatile components, and samples that have large mismatch of particle sizes;

- Carefully assess particle-size distributions of mixtures as particle size affects most measurements:
- Recognize that relative sensitivity to a standard can change when testing conditions are altered because the standard may behave differently to the altered conditions than the target material;
- Recognize that SSST testing may not reflect the true sensitivity of the material for a specific appli-

cation because of the dependency of the results on experimental configuration;

- Develop new methods to test liquids, specifically dealing with volatility issues and development of an appropriate liquid standard;
- Develop instrument-based detection to lesson reliance on observation.

Although further research to advance a better understanding of SSST testing on a fundamental level is slow in being funded, efforts to better standardize testing procedures are being forwarded by the Explosives Testers User Group, organized by Safety Management Services, which is an organization that includes many different energetic materials testing laboratories. <sup>18</sup>

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#### References

- 1. Good general description of small safety and thermal testing of explosives can be found at <a href="http://en.wikipedia.org/wiki/Safety\_testing\_of\_explosives">http://en.wikipedia.org/wiki/Safety\_testing\_of\_explosives</a>.
- 2. DOE Manual 440.1-1A, "DOE Explosives Safety Manual," **2006**, January 9, <a href="http://www.directives.doe.gov">http://www.directives.doe.gov</a>.
- 3. Sandstrom, M. M., Brown, G. W., Preston, D. N., Pollard, C. J., Warner, K. F., Sorensen, D. N., Remmers, D. L., Phillips, J. J., Shelley, T. J., Reyes, J. A., Hsu, P. C., and Reynolds, J. G., "Inter-laboratory Comparisons of Results from Small-Scale Safety and Thermal Testing of Improvised Explosives" *Propellants Explos. Pyrotech.*, accepted for publication 2014.
- 4. Reynolds, J. G., Sandstrom, M. M., Brown, G. W., Warner, K. F., Phillips, J. J., Shelley, T. J., Reyes, J. A., and Hsu, P. C., "DHS Small-Scale Safety and Thermal Testing of Improvised Explosives—Comparison of Testing Performance" *J. Physics, Conference Series* **500**, 1-6, 2013.
- 5. Sandstrom, M. M., Brown, G. W., Warner, K. F., Sorensen, D. N., Remmers, D. L., Whinnery, L. L., Phillips, J. J., Shelley, T. J., Reyes, J. A., Hsu, P. C., and Reynolds, J. G., "Integrated Data Collection Analysis (IDCA) Program—SSST Testing Methods" *IDCA Program Analysis Report* **009**, LLNL-TR-630173 (742792), March 25, 2013; available through Lawrence Livermore National Laboratory Technical Information Center.
- 6. Olinger, B. D., Sandstrom, M. M., Brown, G. W., Warner, K. F., Sorensen, D. N., Remmers, D. L., Moran, J. S., Shelley, T. J., Whinnery, L. L., Hsu, P. C., Whipple, R. E., Kashgarian, M., and Reynolds, J. G., "Integrated Data Collection Analysis (IDCA) Program—Mixing Procedures and Materials Compatibility" *IDCA Program Analysis Report* **002**, LLNL-TR-422028, December 27, 2009.
- 7. Olinger, B. D., Sandstrom, M. M., Brown, G. W., Warner, K. F., Sorensen, D. N., Remmers, D.

- L., Moran, J. S., Shelley, T. J., Whinnery, L. L., Hsu, P. C., Whipple, R. E., and Reynolds, J. G., "Integrated Data Collection Analysis (IDCA) Program—Drying Procedures" *IDCA Program Analysis Report* **004**, LLNL-TR-465872, April 27, 2010.
- 8. Dixon, W. J., and Mood, A.M., "A Method for Obtaining and Analyzing Sensitivity Data" *J. Am. Stat. Assoc.*, 43, 109-126, 1948.
- 9. Department of Defense Ammunition and Explosives Hazard Classification Procedures, TB 700-2 NAVSEAINST 8020.8B TO 11A-1-47 DLAR 8220.1, January 5, 1998.
- 10. Warner, K. F., Sandstrom, M. M., Brown, G. W., Remmers, D. L., Phillips, J. J., Shelley, T. J., Reyes, J. A., Hsu, P. C., and Reynolds, J. G., "ABL and BAM Friction Analysis Comparison" *Propellants Explos. Pyrotech.*, submitted 2014.
- 11. Brown, G. W., Sandstrom, M. M., Preston, D. N., Pollard, C. J., Warner, K. F., Sorensen, D. N., Remmers, D. L., Phillips, J. J., Shelley, T. J., Reyes, J. A., Hsu, P. C., and Reynolds, J. G., "Statistical Analysis of an Inter-laboratory Comparison of Small-Scale Safety and Thermal Testing of RDX" *Propellants Explos. Pyrotech.*, accepted for publication, 2014.
- 12. Neyer, B. T., "D-Optimality-Based Sensitivity Test" *Technometrics*, 36, 48-60, 1994.
- 13. Devore, J. L., *Probability and Statistics for Engineering and the Sciences*, 8th ed., Brooks/Cole pub., Boston, MA, **2012**.
- 14. ANOVA analysis performed by commercial software; Minitab Inc., 1829 Pine Hall Rd, State College, PA, USA 16801; <a href="www.minitab.com">www.minitab.com</a>.
- 15. Fisher, R. A., *Design of Experiments*, Oliver & Boyd, London, **1935**.
- 16. Reynolds, J. G., Sandstrom, M. M., Brown, G. W., Warner, K. F., Shelley, T. J., and Hsu, P. C., *Challenges of Small-Scale Safety and Thermal Testing of Improvised Explosives*, Presentation Pacifichem, Honolulu, HI LLNL-PRES-461618 **2010**, December 17.
- 17. See, for example, Bowden, F. P., and Gurton, O. R., "Initiation of explosives by impact and fric-

- tion: the influence of grit" *Proceedings of the Royal Society London A*, 198 (1054), 337-349, 1949.
- 18. The Explosives Testers User Group is organized by Safety Management Services, West Jordon, UT. For further information contact Robert Ford at RFord@smsenergetics.com.

# Questions and Answers from the review:

Question 1: The paper begins with a discussion of the differences between sensitively results between the various testing labs. The descriptions are very qualitative (e.g. LANL tends to be more sensitive). It would be nice to give a more quantitative description of this (e.g. LANL tended to show sensitive that were XX-YY% greater than the average).

Answer 1: The more quantitative view of the proficiency test is presented in reference 11. Without knowing the statistical significance of differences between values from each laboratory, comparing the differences in results as a percentage would be confusing and possibly misleading. Reference 11 frames the differences in context of testing among the participants of the proficiency test, as well as what would be expected if any laboratory were doing the testing.

Question 2: In the section comparing the BAM and ABL friction tests, the paragraph beginning with "A better indicator of the differences..." begs for more clarification. Why are these relative sensitivities a better indicator of the differences between each method?

Answer 2: The application of force by BAM is a weight on a pin that is applied to the sample staged on a ceramic plate. The application of force by ABL is the force of a pendulum at a specific velocity (which can be varied) colliding with a steel plate that has a stationary grooved wheel on top of the sample. Although the energy transferred in both cases can probably be calculated or measured, the application of the energy is by completely different mechanisms. Figure 4 diagrams this in the drawings. To adequately compare the absolute numbers would require engineering very significant engineering models of both systems. Empirically, we tried to see if there was a correlation, but the paper summarizes that there was not. The net

level is to compare the *relative* ordering. Added to the text: The relative ordering of the  $F_{50}$  or TIL values of the sensitivities of the materials highlight the differences (and similarities) of the two testing methods. If the sensitivity of a specific material is compared relative to a well-characterized standard, then the almost impossible task of comparing sensitivities in kg to sensitivities in psig at a specific velocity is somewhat overcome.

Question 3: In the section on the statistical analysis of RDX you suggest that the ARL 180 data is the outlier. Is there a reason/hypothesis for this? You touch on some of the variables that could cause this in the next section, but never directly address this issue.

Answer 3: This is not true. The paragraphs that follow this statement directly address this issue—the application of ANOVA analysis as well as subgrouping by Tukey and by Fisher methods. I reworded the statement following to assist the reader to recognize the effort we put into performing statistical evaluations to make these statements.

Question 4: In the section on correlation with test-

ing variables, you mention possible correlations with sandpaper and humidity. These statements need more clarification as to the nature of these correlations or what is being done to understand them.

Answer 4: Reference 3 shows many of the effects of sandpaper type on impact sensitivities for HMEs. The effect of grit on non-shock initiation has been studied for 75 years and is still not completely understood. Humidity experiments will not be studied further in this proficiency test. Text has been added to explain this, as well as additional references.

Question 5: The conclusions section talks about proposed changes to SSST to improve reliability of the tests. Is there an effort to develop standards to address the issues identified? If, so I think it would be good to mention that work.

Answer 5: Added text and reference to the SMS ET User group that currently is organizing a round robin test that will help standardize testing procedures as well as instrument calibrations and incorporation of other tests.

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